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SENSORS AND MICROSYSTEMS

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SENSORS AND MICROSYSTEMS

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FOREWORD

This volume gathers the papers presented at the Sixth Italian Conference on Sensors and Microsystems, held in Pisa 3-5 February, 2001, and hosted by the Scuola Superiore Sant'Anna. The Conference was jointly organized by Italian Association for Sensors and Microsystems (AISEM) and the Scuola Superiore Sant'Anna.

As for the previous editions, this book of proceedings offers a view of the current Italian research and development in the wide fields of Sensors and Microsystems; the variety of topics and the quality of papers offer all readers an opportunity to get an insight about the research status in Italy. This proceedings contains selected contributions from 37 different institutions operating in Italy both at academic, public and private research institutions.

Many aspects of Sensors and Microsystems related disciplines are covered in this book, ranging from material science to complete applications and multifunctional systems.

Before the Conference a two days school on fundamentals in Sensors and Microsystems science has been organized. The fruitful results of this initiative have suggested the idea to repeat the experience at next conference.

During the Conference, Prof. Emilio Gatti, Polytechnic of Milan, was honoured by a special AISEM prize for its brilliant scientific career and for its pioneering work on sensors.

Finally, we would like to express our deepest thanks to the staff at the Scuola Superiore Sant'Anna for its brilliant work in this important event.

Corrado Di Natale
Arnaldo D'Amico
Paolo Dario

particles in the 90÷115 degrees range should be noted. This means that the system is potentially capable of reproducing the shape of the scattering diagram associated with a given particle size, with a sufficient resolution to allow its typical features, such as the location of a flex or minimum point, or the sign of derivative in a particular interval, to be identified.

Conclusions

We showed that our polar nephelometer has sufficient resolution for reproducing correctly the features of the scattered light angular distribution for a monodisperse particulate. Because these are characteristics of their dimension, it is possible to extrapolate particle dimensions from scattering data, by means of a suitable model. This task can be accomplished easily by using an Artificial Neural Network [5]. The next step will attempt to extend this procedure to polidisperse particulate, which will be more difficult since averaging on different diameters tends to smooth scattering diagram features. However the possibility of choosing different fiber-GRIN combination will allow us to compensate for this effect by increasing the angular resolution, thus making this instrument a very flexible tool for the study of suspended particles.

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ANTI-BIOFOULING COATINGS FOR OPTICAL FIBER SENSORS

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Abstract

One of the most relevant problems of using optical fiber sensors in real-world environments is surface fouling, that is, the cumulative build-up of undesirable material on the working surface of the sensor.
The present paper presents the results of tests of anti-biofouling coated fiber optic probes for reflectance spectroscopy in blood-simulating 'foul' media, namely Bovine Serum Albumin (BSA) and Fibrinogen. The anti-biofouling coating was a cross-linkable polymer with silane functionality, to improve adhesion to silica-containing substrates.

Introduction

A major cause of loss or failure in sensor performance in medical or environmental applications is the growth of biological material coming to live in contact with a wide variety of surface materials. Where fouling can occur the use of many otherwise excellent sensors is precluded.
In particular, sensors of physiological parameters[1] represent one of the most important aspects of biomedical instruments, and their use may result in better diagnoses and a better quality of health care. An accurate description of the functions of physiological systems and organs is performed in vivo by means of direct observations and measurements of a number of fundamental variables. For medical applications, a distinction must be made between invasive (e. g. in the blood-stream or in tissue) and non invasive (e. g. on the skin) measurements. Clearly, there is a preference for non-invasive measurements in view of patient

safety and comfort. However, the necessity for more accurate and reliable evaluations of the measured physiological parameters, may require the use of invasive sensors. Hence, miniaturization is essential for invasive sensors, while the bio-compatibility of the materials is important[2]. Mechanisms that can give rise to problems include hemodialysis, likewise plaque aggregation and coagulation[1,3].
This paper discusses the anti-biofouling capability of a polymer coating for optical sensors when these are exposed to blood-simulating fluids, such as Bovine Serum Albumin (BSA) or Fibrinogen, in a dynamic environment.
Optical tests on coated and un-coated probes performed during long-term exposure, together with comparisons of their results, provide a means for understanding not only the anti-biofouling capability of polymer coating, but also for evaluating whether the fouling occurs and when it occurs in both coated and un-coated probes.

Polymer coating

Many materials used in the medical device industry were not originally developed for these applications. In general, these materials elicit adverse biological responses when in contact with body fluids such as blood, and the cascade of events when blood interacts with an artificial surface are well characterised. Protein adsorption, platelet adhesion, and activation of the coagulation pathway can subsequently lead to thrombus formation, with grave clinical consequences in the absence of anticoagulant. In recent years, various approaches for overcoming these problems by improving the bio-compatibility of materials have been advocated. One approach is that of bio-membrane mimicry, whereby the surface of a material is coated with a derivative of Phosphorylcolina (PC). The PC anti-biofouling coating used by probes presented in this paper is a proprietary invention of Biocompatibles Ltd.
PC is the major lipid head-group component found in the outer surface of biological cell membranes. The application of PC coatings to a range of materials has been discussed, and the surfaces using in vitro bio-combatibility tests have been characterised[4]. Studies of fibrinogen adsorption, platelet binding, and bacterial adhesion have shown significant reductions in the adsorption of these components to various PC-coated materials relative to un-coated controls. Materials tested, among others, include PVC, polyethylene, policarbonate, and nylon.
The stability of PC coatings has been studied using radio-labeled derivatives. Results of the use of several materials show that physiabsorbed PC coatings are extremely stable, thus making the coatings suitable for use in a wide variety of medical applications. Extensive biological evaluations to assess the toxicological profile of PC polymers and coated devices have also been carried out, in all tests, the materials have been shown to be non-toxic, thus making them suitable for human use. The PC polymers must adhere to specific optical substrates, i. e. glass and silica, and must produce a biocompatible interface. This PC biocompatible interface then has the potential to reduce the amount of biological fouling that can occur on these surfaces. The coating must have sufficient durability to remain on the surface and, for optical sensors, the coating must not interfere with the optical properties of the device.
In particular, between all PC coatings prepared, the best results have been obtained using the PC1036 polymer, designed with 23% wt PC with a hydrophobic film-forming monomer and a silane functional cross-linker. Biocompatibilty tests of it have shown a 76% reduction in Fibrinogen adsorption (relative to un-coated controls) and an 81% reduction in E. Coli adhesion (relative to un-coated controls).
PC1036 has the added benefit that the silane functionality has the potential to interact chemically with surface Si-OH groups on glass and silica, thus making possible even greater stability.
The optical properties of the polymer are very encouraging. Polymer has very little absorption in the useful part of the UV/VIS spectrum (250nm upwards) and only specific IR absorptions. These IR absorptions occur between 500-4000 cm^{-1}, and are likely to be very similar for all of the PC polymers as they are predominantly due to the residual methacrylate functionality, that is common to all the PC polymers produced.

Fiber optic probes

The basic optical fiber probe is outlined in Figure 1. A pair of identical optical fibers are inserted and then glued together, inside a plastic holder. A light-diffusing plastic cap containing a glass disc is placed in front of the fiber ends by means of a spring. The optical fiber holder is threaded in order to optimize the cap positioning with respect to the fibers, by simply screwing or unscrewing the spring.
In practice, when the probe is immersed in a liquid, the liquid flows between the fiber ends and the glass disc, coming in contact with both of them. If the two fibers are coupled respectively to a source and to a detector, a transmission measurement is carried out and the signal detected gives an optical measurement of liquid-induced fouling of the fiber ends and the glass.
The optical fiber probe treated with anti-fouling coating is a basic probe in which the fiber ends and the glass disc are coated with an anti-fouling layer, as oulined in Figure 2.
The performance of coated and un-coated probes in environmental conditions can be controlled by immersing both probes in the same medium while optically exciting the illumination fibers and continuously detecting the back-transmitted signals. A comparison of the back-transmitted signals indicates the effectiveness of the anti-fouling coating.

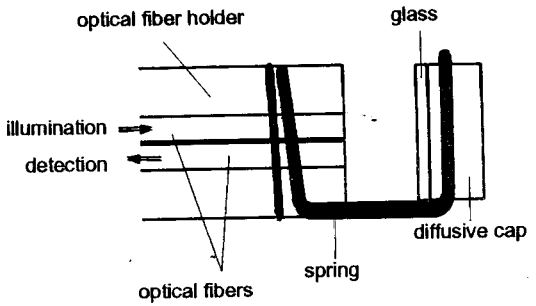


Figure 1. Optical fiber probe: basic design

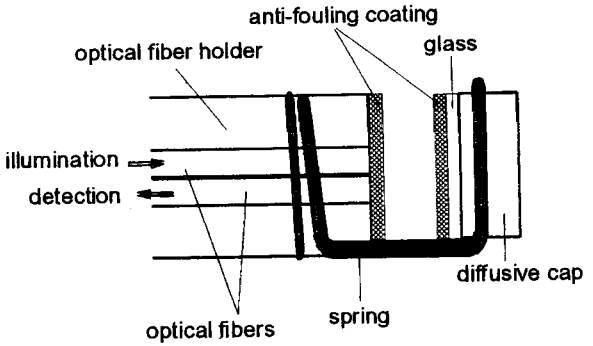


Figure 2. Optical fiber probe coated with anti-fouling PC layers

Table I summarizes the materials used for implementation of the optical fiber probe. The optical fibers and the glue are commericially available, while the other components have been fabricated at IROE-CNR.
The dimensions of the optical fiber holder are: 3.0 mm diameter, 7.0 mm length. Total dimensions of the probe, including spring and cap are: 4.0 mm diameter, 13.0mm length, including the glue.

Table I. Materials used for implementing the optical fiber probe

Component	*Material*
Optical fibers	3M-TECS: FT-200-EMT, core diameter: 200 μm, clad diameter: 230 μm, jacket diameter: 500 μm
Optical fiber holder	Nylon
Cap	Moplen
Glass	Slide for microscopy, Cole-Palmer #E-48500-00
Glue	Norland adhesive #61, UV curing
Spring	Stainless steel

Experimental setup for optical tests

A scheme of the experimental set up to control the performance of probes in environmental conditions is illustrated in Figure 3. Coated and un-coated probes are immersed in a by-pass flow cell that is connected in series with the reservoir containing the liquid under test and with a peristaltic pump.

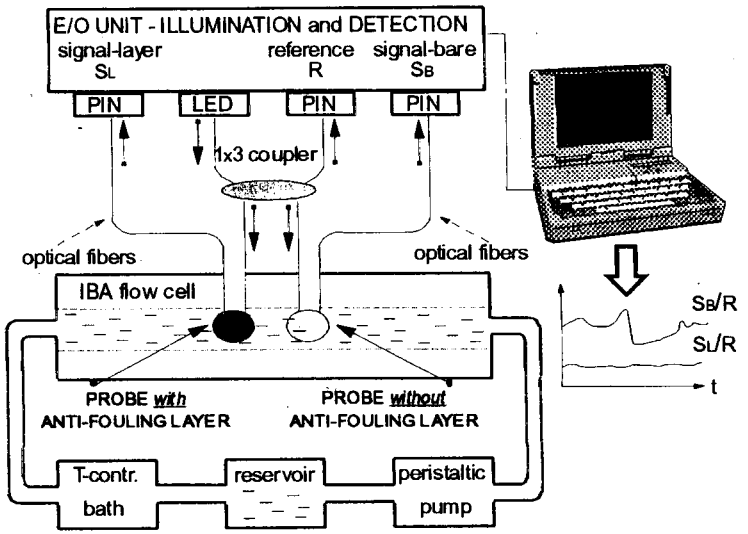


Figure 3 Scheme of the experimental setup for fiber optic probe testings

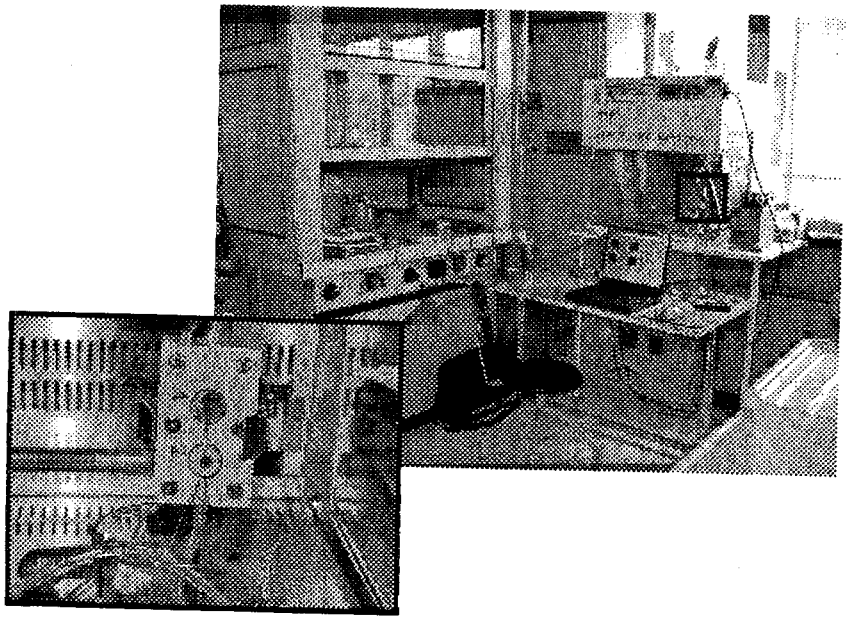


Figure 4 Experimental setup for optical tests of PC1036 anti-fouling coated and un-coated fiber optic probes

The probes are connected to a PC-interfaced electro-optic unit which provides illumination, detection and data processing.
A large core optical fiber is connected to a LED and is split by a 1 x 3 coupler into three channels: one optical fiber is connected directly to a photodetector, providing a reference signal, while the other two fibers are connected respectively to the illimination fibers of the probes under test. The detection fibers of the probes are directly connected to the remaining two photodetectors. Signals coming from the probes are referenced to the LED signal, in order to avoid taking into account possible LED fluctuations.
The electro-optic unit is driven by a Virtual Instrument for LabVIEW® which provides data logging and display.

Experimental results of optical tests of PC1036 anti-fouling coated and un-coated fiber optic probes

Fiber optic probes were tested according to the following test rig:

a) two identical fiber optic probes were considered, one of which was PC1036-coated;
b) both probes were optically excited, and the signals were continuously detected while immersing the probes in the same 'clean' or 'foul' medium; long-term tests of more than 20 hours were performed;
c) a visual check of both probes was performed using optical microscopy.

Tests in 'clean' media were aimed at checking the photo- and mechanical stability of the coating. The following 'clean media' were considered:

- Normal saline;
- Glucose solution;
- Ringer Acetate;
- Solution of electrolytes and glucose;
- Emulsion of micronized lipids and phospholipids;
- Solution of amminoacids and electrolytes;
- Normal saline and vister-based anticoagulant solution, for verifying the effects of an anticoagulant additive;
- Solution of amminoacids and electrolytes and purified human albumin.

These tests showed good mechanical stability for the PC1036 coating, In particular, the last test, in which human albumin was added to a 'clean' solution, showed little fouling of the un-coated probe. The fouling was confirmed by means of a microscopic check.

Tests in 'fouled' media' were performed by using

1. Bovin Serum Albumin (BSA) in 0.9% NaCl solution, BSA 1 g/l.
2. Fibrinogen in 0.9% NaCl solution, 0.1 g/l.

The test procedure was as follows:

- Before all measurements, a photostability test over 20-24 hours was carried out in order to check the photostability of the anti-fouling coating. Optical signal variation within ±1%, of the same order of electronic noise, was typically measured, demonstrating excellent photostability of the coatings.
- All solutions were sterile, and were handled inside the laminar flow hood so as to guarantee satisfactory sterility.
- Flow cell, glasses, and flow tubes were sterilized before each test.
- The fiber optic probes were not sterilized.

Results of measurements

After each test, the anti-fouling coated probe was visually checked by disassembling the probe and by cheking the image of the fiber optic pair in the microscope.
Tables II and III show the results of probe optical exitation, together with the images of the microscopic checks. These results were obtained by means of tests performed over a minimum period of 24 hours.

Table II: PC-1036 coated and un-coated optical fibers tested in a by-pass flow cell in BSA-Bovine Serum Albumin 1g/l in Normal Saline (0.9% NaCl Solution)

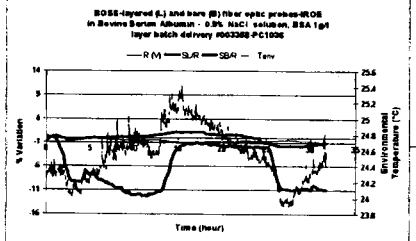
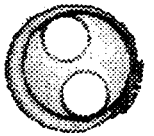
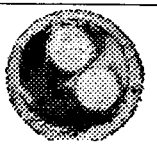
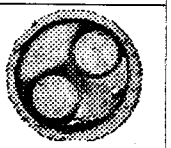
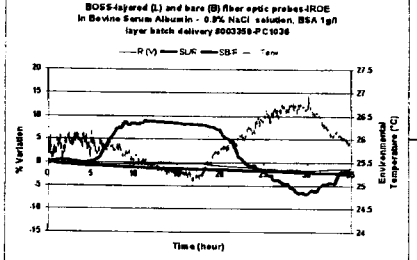
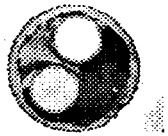
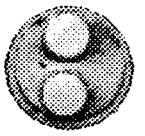
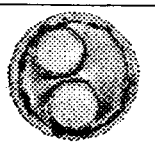
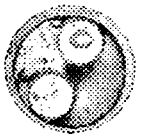
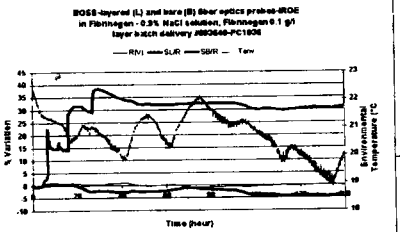
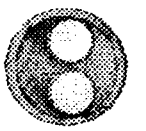
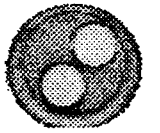
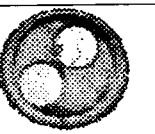
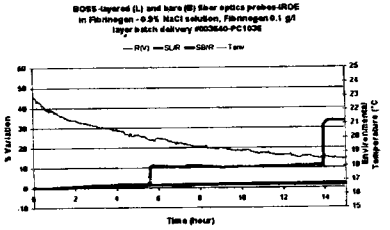
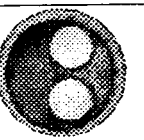
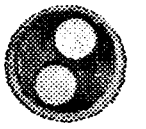
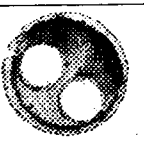
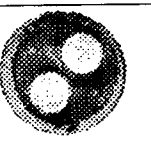
% variation of detected signals from optical fiber probes as a function of time	images of fibers before (§) and after (§§) immersion in foul medium	
	coated	un-coated
	 §	 §
	 §§	 §§
	 §	 §
	 §§	 §§

Table III: PC-1036 coated and un-coated optical fibers tested in a by-pass flow cell in Fibrinogen 0.1g/l in Normal Saline (0.9% NaCl Solution)

% variation of detected signals from optical fiber probes as a function of time	images of fibers before (§) and after (§§) immersion in foul medium	
	coated	un-coated
	 §	 §
	 §§	 §§
	 §	 §
	 §§	 §§

Tests in BSA showed excellent results as far as both optical signal behaviour and microscopic checks are concerned. The signal variations of un-coated probes were in the ± 10-30% range, while coated probes exhibited a nearly stable optical signal. This excellent result was confirmed by microscopic checks, in which adhesions in non-coated fibers appeared.
Tests in Fibrinogen were also good. The signal variations of un-coated probes were in the ± 20-35% range, while coated probes exhibited a nearly stable optical signal.

Conclusions

Optical fiber probes coated by means of an anti-biofouling layer have been tested in blood-simulating 'foul' media.
Blood is made of plasma plus cellular elements[5]. The plasma is the liquid part of the blood: it is composed by many proteins, but the most representative ones are Albumin (55-69% min-max value), Globulin (26-50% min-max value) and Fibrinogen. The results obtained are encouraging, because they were obtained with the most representative blood proteins, i. e. BSA and Fibrinogen.

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DEVELOPMENT OF A CMOS ASIC FOR SMOKE DETECTION

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We report on an ASIC in 0.8 μm CMOS technology especially taylored for smoke detection applications. The chip consists of an integrated photodiode, which reveals the light scattered by smoke particles, and of the related electronics. The circuit design, based on the *Switched-Capacitors* technique, accomplishes low noise specifications by means of an accurate design of the first read-out stage and by employing Correlated Double Sampling filtering blocks. Preliminary results from the electrical characterization of the first chip prototype allowed the basic design methodology to be validated.

1 Introduction

Most of commercially available smoke detectors make use of optical sensors exploiting the scattering principle. In these devices, the radiation provided by an IR-LED source illuminates the smoke-detection-chamber, which allows the entry of smoke particles while shielding from the ambient light. The presence of smoke is detected by a photosensor which reveals the light scattered by smoke particles. These systems are normally based on discrete components, including a large, high-efficiency photodiode, which is necessary to detect the very low photocurrent associated with the radiation scattered by the smoke cloud using conventional read-out circuits. The main drawbacks of this approach are the cost of components assembly and of high performance photodiodes and the demand of a preliminary calibration, as required by the high sensitivity of circuit characteristics to components tolerance. In alternative, a monolithic CMOS approach could provide several advantages, including easier assembly, better noise rejection, higher reliability and lower cost, and, in perspective, could also ease the on-chip implementation of enhanced functionalities like compensation and self-calibration.

In this work we report on the development of an ASIC for smoke detection, fabricated in 0.8 μm CMOS technology. The circuit design is based on

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